ORTIZ POLYMER MECHANICS LABORATORY MANUAL

Room 12-065 (Chemistry Lab, Phone : (617) 258-6154)
Room 13-5037 (Nanomechanics Lab, Phone : (617) 253-8779)
Massachusetts Institute of Technology
Department of Materials Science and Engineering
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Phone : (617) 253 6819 **Fax** : (617) 258 6936 **Email** : tbdoyle@mit.edu

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1. CHECKLIST FOR NEW GROUP MEMBERS AND COLLABORATORS

ITEM TO BE COMPLETED	DATE COMPLETED
1. Please read this entire laboratory manual.	
2. Please obtain copies of the AFM and MFP instrumentation manuals. Please obtain, read, and go through the DI Scanning Probe Microscopy Training Notebook (all can be obtained from Tim Doyle in 12-009).	
3. Finish all safety trainings as outlined in Section 6. of this laboratory manual.	
4. Collaborators : please read, sign, and return User Collaboration Agreement Form to Prof. Ortiz.	
5. Please see Tim Doyle to obtain keys to the labs and photocopy cards (primary group members only).	
6. Ask Professor Ortiz for a research account number and inform Tim Doyle of this number.	
7. Obtain a laboratory notebook from the chemical stockroom.	
8. Set up date for AFM/ MFP training : AFM trainer : Laurel Ng (ljng@mit.edu) MFP trainer : Monica Rixman (mrixman@mit.edu)	

2. GENERAL LABORATORY POLICIES

1. Ortiz primary students and postdocs have priority on all laboratory equipment.
2. All new group members and collaborators should be initially trained by an Ortiz graduate student (MFP trainer : Monica Rixman (mrixman@mit.edu), AFM trainer : Laurel Ng (ljng@mit.edu)) The trainer should be provided with a one-page summary of their planned experiments in the near future before beginning training. Observation of experiments performed by another experienced user multiple times is strongly suggested.
4. Safety precaution sheets should be read carefully and adhered to before starting any experiments.
5. Any problems with the equipment should be reported immediately to Prof. Ortiz.
6. Researchers should reserve time on the equipment by signing the appropriate schedule. It is suggested to sign out either morning/ afternoon/or evening block, not more than one full day at a time.
8. All students must sign and date the log-books for the equipment each time they use it. Prof. Ortiz will review the log books on a weekly basis.
9. Collaborators should keep track of and pay for all of the supplies they use, i.e. cantilever / probe tips. The collaborating students advisors are expected to take care of the costs for chemicals, materials, and supplies related to the project.
10. All group members are expected to attend the weekly Ortiz group meeting (Mondays 5pm, MIT 12-009) and informally present what they have done during the week.
11. All laboratory notebooks and keys should be returned to Tim Doyle upon completion of the research project and work in the lab.
12. Abstracts for talks, papers, and any other published work resulting from experiments done in the Ortiz Lab should be sent to Professor Ortiz and all other authors at least one week before the deadline.
13. Always close and lock all doors to laboratory equipment.
14. No laboratory equipment should be removed from the lab without prior permission from Prof. Ortiz and verification with the other laboratory group members.

3. ORTIZ LAB PHONE LIST F01

CHEMISTRY LAB : 12-065 , **PHONE :** 258-6154

NANOMECHANICS LAB : 13-5037 , **PHONE :** 253-8779

GRAD STUDENT OFFICE : 12-022 , **PHONE : 258-5934**

PRIMARY GROUP MEMBERS :

Name	Email (@mit.edu)	Office / Work Phone (area code : 617)	Phone (Home)	B-DAY	Collaborating Group
Rafael Bras (UGRAD-DMSE-YR 4)	rebras	RM 12-022 / 258-5934	(617) 232-3257		**Alex van Oudenaarden** (avano@mit.edu) (Physics-MIT, 253-4446, 13-2010) & **Arpita Upadhyaya** (arpitau@mit.edu, 253-4829, RM 13-2054)
Patty Chen (UGRAD-DMSE-YR 3)	ppchen	RM 12-022 / 258-5934	225-9491		**Judith Stein** steinj@crd.ge.com **Christina Darkangelo Wood** : woodch@crd.ge.com GE CRD/ Polymer Materials Laboratory 1 Research Circle Bldg. K-1 Rm. 4a54 Niskayuna, NY 12309 (518) 387-5136
Delphine Dean (EECS-PHD-YR1)	finou	RM 38-377: 253-8385	497-1283	May 11	**Alan Grodzinsky** (EECS/BEH/ CBE-MIT) alg@mit.edu, 253-4969
Tim Doyle (Administrative Assistant)	tbdoyle	RM 12-009 / 253-6819 Fax : 258 6936			
Mariselma Ferriera (Vistiing GRAD)	mari@cnpdia.embrapa.br, mariselmaf@hotmail.com	RM 12-022 / 258-5934			**Dorothy Hosler** (ARCHEOLOGY-DMSE) hosler@mit.edu **Luiz Henrique C. Mattoso** Embrapa Instrumentação Agropecuária

					Rua XV de Novembro, 1452 Caixa Postal 741 São Carlos, 13560-970, SP, Brasil mattoso@cnpdia.embrapa.br
George Gluck (BIO-YR 4)	ggluck	RM 12-022 / 258-5934	794-7110		**Rich Gilbert** (MECHE-CBE-MIT) : rgilbert@mit.edu cell : 620-2032 pager :546-8940 office (MIT) RM 3-335 : 253-2223
Ryan Jones (GRAD-MECHE-YR2)	jonesre	3-0229			**Douglas Hart / Gareth McKinley** (MECHE-MIT)
Jennifer McKeehan (DMSE-GRAD-YR1)	jenmck				
Laurel Ng (GRAD-BEH YR1)	ljng	RM 38-377: 253-6944	436-5692		**Grodzinsky** Lab (EECS/BEH/ CBE-MIT)
Christine Ortiz (DMSE-Research Advisor)	cortiz	RM 13-4022 / 452-3084 Fax : 452-3085	557-9996 407-4741 (cell)	Nov 27	
Monica Rixman (GRAD-DMSE-PPST-YR2)	mrixman	RM 12-022 / 258-5934	629-5944 (cell) 549 4512	Nov 15th	
Joonil Seog (GRAD-MECHE-PPST YR4)	jseog	RM 12-022 / 258-5934 and RM 38-377 / 253-8385	577-5699	Sept 18th	**Grodzinsky** Lab (EECS/BEH/CBE-MIT), Sasisekharan Lab (BEH-MIT)
Kuangshin Tai (DMSE-GRAD-YR1)	taik		225-9584		
D. Andrew Wilkinson (UGRAD-MECHE-YR3)	awilkins		621-0630		**Rich Gilbert** (MECHE-CBE-MIT) : rgilbert@mit.edu cell : 620-2032 pager :546-8940 office (MIT) RM 3-335 : 253-2223
Dong Zhang (POSTDOC-CHEMISTRY)	dozhang@yahoo.com				

COLLABORATORS :

Name	Email (@mit.edu)	Office / Work Phone (area code : 617)	Phone (Home)	B-DAY	Collaborating Group
Reuben Dormike	rdomike	617 253-0472	617-794-4441		**James Cooney** (CHEME cconey@mit.edu, 617 253-3108
Xueping Jiang (GRAD-CHEME YR2)	xpjiang	RM 66-525 253-6450	577-5637		**Paula Hammond** (CHEME-MIT) hammond@mit.edu, 258-7577, RM 66-550
Ryan Jones (GRAD-MECHE-YR2)	jonesre	3-0229			**Douglas Hart / Gareth McKinley** (MECHE-MIT)
Sang Park (DMD)	sang_park@hsdm.harvard.edu	432-1472	617-290-8136		**Juan Loza** (Harvard Dental School) : juan_loza@hms.harvard.edu, 432-4252
Winnie Yong (GRAD-YR2-NUCLEAR) Brice Smith (GRAD-YR2-PHYSICS)	wyong elrond		WY (225-1163) BS(354-4827)		**George B. Benedek** (PHYS-MIT, benedek@mit.edu)

4. COLLABORATOR USER AGREEMENT AND POLICIES

ORTIZ POLYMER MECHANICS LABORATORY

COLLABORATOR USER AGREEMENT & POLICIES

Supervisor: Christine Ortiz, Assistant Professor
Department of Materials Science and Engineering (DMSE)
Room 13-4022, 77 Massachusetts Avenue
Cambridge, MA 02139 USA
Phone : (617) 452 3084 *Fax* : (617) 452 3085
Email : cortiz@mit.edu *WWW* : http://web.mit.edu/cortiz/www/
Assistant : Tim Doyle *Office* : Room 12-006
Phone : (617) 253 6819 *Fax*: 617) 258 6936 *Email* : tbdoyle@mit.edu
Labs: *Chemistry*: RM 12-065, Phone : (617) 258 6154
Mechanics : RM 13-5037, Phone : (617) 253 8779

General Policy: The lab welcomes collaborators participating in research projects of mutual interest, but can not be employed in any way as a standard service facility (i.e. the DMSE Center for Materials Science and Engineering (CMSE) and Center for Biomedical Engineering (CBE) can provide equipment for such work). Hence, to avoid conflicts collaborating students are expected to adhere to the following guidelines.

Specific Policies:

1. The collaborating student / post-doc should write up a one-page plan of experiments to be done in the lab and have them approved by C. Ortiz before beginning work. An additional one-page research plan is expected at the beginning of each semester.
2. Ortiz graduate students have priority on all equipment.
3. Collaborating students / post-docs should be initially trained by an Ortiz graduate student. The collaborator should provide the trainer with the one-page summary of work he or she would like to accomplish prior to the training. Observation of experiments performed by another experienced user multiple times is strongly suggested.
4. Safety precaution sheets should be read carefully and adhered to before starting any experiments.
5. Any problems with the equipment should be reported immediately to Prof. Ortiz.
6. The collaborating student / post-doc and their doctoral / post-doctoral advisor are responsible for any damage to the equipment which is found to be at the fault of the student. The student is responsible for arranging quick repair of any damage they may have caused.
7. Researchers should reserve time on the equipment by signing the appropriate schedule. It is suggested to sign out either morning/ afternoon/or evening block, not more than one full day at a time.
8. All students must sign and date the log-books for the equipment each time they use it. Prof. Ortiz will review the log books on a weekly basis.
9. Collaborators should keep track of and pay for all of the supplies they use, i.e. cantilever / probe tips. The collaborating students advisors are expected to take care of the costs for chemicals, materials, and supplies related to the project.
10. Joint publication is expected for research conducted in the laboratory. If an Ortiz graduate student is integrally involved with the research, it is expected that they will maintain coauthorship on the publication as well.
11. If a collaborating student / post-doc has performed experiments during a particular week, it is expected that they attend the following week's Ortiz group meeting (Monday 5pm, MIT 12-009) and informally present what they have done.

O R T I Z P O L Y M E R M E C H A N I C S L A B O R A T O R Y

C O L L A B O R A T O R U S E R A G R E E M E N T F O R M

Collaborator Name: _____

Campus Address: _____

Campus Phone / Email: _____

Home Address: _____

Home Phone / Email: _____

Advisor: _____

Advisor Address: _____

Advisor Phone / Email : _____

Expected Dates of Experiments and Lab Usage: _____

Date of AFM Training: _____

AFM Trainer: _____

Date of MFP Training: _____

MFP Trainer: _____

STUDENT SIGNATURE : _____

ADVISOR SIGNATURE : _____

ORTIZ SIGNATURE : _____

Thank you for your cooperation with this policy. It is intended to allow our group to keep track of how our equipment is being used and to attempt to insure quality control on equipment usage and safety procedures. You will be provided with a copy of the signed form.

5. LABORATORY SUPPLIES ORDER FORM

ORTIZ LABORATORY SUPPLIES ORDER FORM

DATE	
VENDOR NAME	
VENDOR TELEPHONE OR URL	
ITEM NAME	
ITEM NUMBER	
PRICE ***(IF OVER $200 OBTAIN AUTHORIZATION FROM C. ORTIZ)***	
QUANTITY	
UNIT ***(e.g. gallon, pack of 12, etc.)***	
DATE NEEDED	
ACCOUNT NUMBER	
ADDITIONAL COMMENTS	

Please send to Tim Doyle (tbdoyle@mit.edu) or drop off to Room 12-099. Call if you have questions (3-6819).

6. LABORATORY SAFETY PROCEDURES AND TRAINING

Manditory :

I. Center for Materials Science and Engineering Requirements (CMSE) :

I.A. Read the OSHA-mandated document, the **Chemical Hygiene and Safety Manual.** You can obtain a copy from the CMSE office, RM. 13-2090.
I.B. Take the Center for Materials Science and Engineering (CMSE) **Chemical Hygiene Hypercourse** at : *http://www.boston-consortium.org/sub_gro_ehs_wbt.shtml*

II. Department of Materials Science and Engineering Requirements (DMSE) :

II.A. Read the **DMSE Department Safety Primer** (printed out in this manual following) :
http://web.mit.edu/course/3/3.041/html/safety/primer.htm
II.B. Read the **DMSE Chemical Hygiene and Safety Plan** manual which can be obtained from Gerald Hughes, DMSE facilities manager; *email*: ghughes@MIT.EDU, *phone*: (617) 452-3938, *address*: 8-309.
II.C. Make an appointment with Fred Wilson (room 13-4078, phone 3-6866) to **take the DMSE safety quiz** on these documents.

All of these items must be completed before keys are given out to the labs. Please inform Professor Ortiz the dates that these are completed.

Advised :

1. Watch the **CMSE Safety Presentation** on videotape.

6.1 The DMSE Safety Primer

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- The DMSE Safety Program
- Emergencies and First Aid
- Basic Elements of Laboratory Safety
- Chemical Hygiene
- Radiation Safety

Electrical safety

- Cryogenic safety
- Fire safety

THE DMSE SAFETY PROGRAM

Introduction

Unfortunately, we tend to think of safety only after an accident. Laboratory safety, however, deserves *and requires* the same planning and attention that we give to research and teaching; it must not be an afterthought. There are both civil and criminal legal consequences of liability; and there are federal, state, and local laws that codify and regulate safe practices and environmental hazards. The details in the regulations that govern safety, environmental hazards, and waste disposal are changing and evolving, but the principles that underlie the approaches to these problems have not changed: *when we are in the laboratory, we must think about what we are doing, we must be aware of the dangers, and we must know what to do if an accident occurs*.

This *Safety Primer* is not intended to be an all-inclusive treatment of laboratory safety; such a document would be extremely large. The *Primer* is condensed from a number of larger documents, and is a summary of fundamental safety procedures all workers must know. It is intended as a first step in what must become a continuing education and concern with safety. Each laboratory situation has its own specific safety rules and procedures, and these must be added to the general information included here. Information in this manual is supplemental to that in other useful sources, such as the *DMSE Chemical Hygiene Plan,* and the *MIT Accident Prevention Guide* .

Responsibilities

Employees, Staff, Students, and Visitors

All students, visiting scientists, employees of DMSE, and all personnel who use the Departmental laboratories are subject to DMSE safety procedures, as augmented by the specific procedures dictated by the individual Laboratory Supervisors (usually the DMSE faculty member in charge of the laboratory). When the laboratory itself does not have a supervisor (true in some central facilities), the worker's advisor or supervisor carries out these duties. Each laboratory user is expected to do the following:

- All laboratory workers, including faculty, students, and visitors must pass a safety test based on this *Primer* before using DMSE laboratory facilities. This test is administered to 3.081 students at the beginning of the term; others should contact the DMSE Safety Technician (Mr. Frederick Wilson, room 13-4078, phone 3-6866) to arrange an appointment.
- The Laboratory Supervisor is required to provide specific information covering the procedures of the particular laboratory involved. Both the worker and the supervisor must sign a form agreeing that this briefing has been carried out satisfactorily before work may begin.

- Access to a laboratory will not be approved, and UROP proposals will not be signed, until the user has passed the examination and provided an acknowledgment of the safety briefing that has been signed by both the user and the Supervisor.
- Workers are to conduct themselves in a safe manner at all times, following the rules outlined in this *Primer* and other appropriate sources of information. *The primary responsibility for safety resides with the individual worker.*
- Workers must insist that coworkers follow safe procedures as well, and should report persistent failure to do so to the Laboratory Supervisor, the Departmental Safety Officer, or the Department Head.
- Workers should notify the Laboratory Supervisor of unsafe procedures in the laboratory, and of means by which they feel safety could be improved.
- Workers must notify the Laboratory Supervisor of *all* accidents, and near-misses as well.

Laboratory Supervisor

Although the individual worker is ultimately responsible for his or her own safety, the Laboratory Supervisor must insure that all laboratory workers have the facilities and training needed to make safe conditions possible. The Supervisor's duties include the following:

- The information in this Safety Primer and the DMSE Chemical Hygiene Plan must be augmented to include written procedures specific to the Supervisor's laboratory. Each laboratory should have an easily located binder containing these "Standard Operating Procedures" and other documents needed to govern safe operating procedures. A Material Safety Data Sheet (MSDS) for each material used or stored in the laboratory must be available in this binder.
- Provide instruction and training to all supervised laboratory workers in safe work practice, in the use of personal protective equipment, and in procedures for dealing with accidents. These briefings must be provided for each laboratory worker before he or she is allowed to work in the laboratory.
- Make certain that the worker has passed the required Chemical Hygiene and Safety Examination. Keys to laboratories or disclosure of lock combinations should not be approved until the worker has passed the examination and has acknowledged the briefing.
- Each laboratory door must have a Green Safety Notice Card displayed on it. This card must contain the name(s) of individuals to contact if a laboratory emergency requires their notifications. The card must be updated whenever new people are assigned to the lab. New cards may be obtained from Frederick Wilson (13-4078/X3-6866) or from the MIT Safety Office.
- Report all accidents to the MIT Safety Office, and work with the Safety Office to improve laboratory procedures so that such accidents are not repeated.
- Define the location of work areas where toxic substances and potential carcinogens will be used, and ensure that the inventory of these substances is properly maintained.
- Define hazardous operations, designate safe practices, and select protective equipment.
- Monitor the safety performance of laboratory workers to ensure that the required safety practices and techniques are being employed.
- Keep the laboratory clear of clutter (unused and obsolete equipment, etc.). Properly dispose of unwanted and/or hazardous chemicals and materials.
- Be prepared to undergo both scheduled and surprise laboratory inspections by MIT and other authorized persons. These may include checks of the physical presence of appropriate safety equipment, presence of readily accessible safety documentation and written procedures, and safety awareness of laboratory workers.

The DMSE Safety and Chemical Hygiene Committee

The Departmental Safety and Chemical Hygiene Committee is appointed by the Head of the Department. The Departmental Safety Officer and the Departmental Administrative Officer are ex-officio members. Currently, the DMSE Safety and Chemical Hygiene Committee consists of:

- Prof. David Roylance, Departmental Safety and Chemical Hygiene Officer
- Mr. Fredrick Wilson, Departmental Safety Technician
- Mr. Patrick A. Kearney

- Mr. Joseph A. Adario

Duties of the Safety Committee include:

- The Committee assists the Departmental Safety Officer in formulating policies and procedures for laboratory safety, and also assists in formulating the examinations required under the Plan.
- Each Departmental laboratory will be inspected twice yearly for compliance with safety procedures; these inspections are supervised by Mr. Kearney.
- The Committee will coordinate safety procedures with the Research Directors for the Center for Materials Science and Engineering (CMSE) and the Materials Processing Center (MPC).
- The Committee will also review accidents and other incidents that involve chemical hygiene and safety.

The MIT Safety Office and Medical Services

The Institute provides a number of central services concerning safety and health. The MIT SafetyOffice (room E19-207, phone 3-4736) and the Environmental Medical Service (Room 20B-238, phone 3-5360) have professional staffs that can be called upon for advice and help on safety and environmental health problems. These staffs offer the following services to the Institute:

- The MIT Safety Office evaluates and implements safety policies and reviews new and existing equipment and operating practices to minimize hazards to the Institute community and visitors from fire, electricity, explosion, pressure, and machinery.
- The MIT Safety Office conducts accident investigations, suggests remedial measures, and administers accident reporting procedures. It also publishes the MIT Accident Prevention Guide, which is available via techinfo on Athena.. In addition, a waste chemical service will pick up potentially hazardous chemicals. Training and assistance in conducting special accident prevention programs are available as required.
- The Environmental Medical Service (EMS) is a unit of the Medical Department. Several health physicists, microbiologists, industrial hygienists, and industrial hygiene engineers (all members of the staff) devote their skills to the protection of the Institute community from radiation, toxic, and biological hazards. All members of the Institute community should feel free to consult with the Environmental Medical Service if they are concerned about the safety of operations involving potential toxic or radiation exposure. A member of EMS is available at any time for assistance in emergencies and can be reached through the Medical Department or Physical Plant Work Control Center.

EMERGENCIES AND FIRST AID

Summoning Help

Every accident is different, and it is not possible to prescribe procedures for responding to them that will work in all instances. But as a general guideline, MIT and DMSE policy is to have the individual laboratory worker perform only minimal emergency actions. When an accident happens that you consider serious and difficult to handle, your response should be to evacuate yourself and others from the scene, and to summon help:

- PULL the FIRE/EMERGENCY ALARM closest to the emergency, and evacuate the Building. Each building has evacuation routes described on cards posted throughout the building, and you must be aware of these routes.
- DIAL 100 from a safe location, give your name, the location of the emergency, and describe the emergency as best you can. Stay on the phone until the police dispatcher hangs up.
- The evacuation instructions specify meeting places at which the building's occupants should gather. Laboratory supervisors should account for the members of their group, to determine if anyone is still in the Building. If you think someone is still inside the building, notify the Fire Marshal/Campus Police/Emergency Response Team.

- After the emergency, promptly report the incident to the MIT Safety Office (phone 3-4736, room E19-207).

Dial 100 for:

- fire
- toxic gas leaks
- Campus Patrol
- ambulance
- serious illness
- accidents

Dial FIXIT (3-4948) for:

- floods
- city gas leaks
- stuck elevators
- loss of heating
- loss of fume hood fans
- lost of electrical power
- loss of ventilation

Other Safety-Related Phone Numbers:

- Roylance, David - DMSE Safety Officer 6-202 3-3309
- Wilson, Frederick - DMSE Safety Technician 13-4078 3-6866
- Diffley, Raymond - MIT Safety Office 3-4736 E19-207
- Clifford, Robert - MIT Industrial Hygiene Office 3-0908 20C-204

Additional Resources:

- Medical Department (24 Hour Emergency) 3-1311 E23-189
- Medical Department 3-4481 E23
- MIT Safety Office 3-4736 E19-207
- Environmental Medical Services 3-5360 20B-238
- Biohazard Assessment Office 3-1740 20C-208
- Radiation Protection Office 3-2180 20C-207

First Aid

In a medical emergency, summon professional medical attention immediately by dialing 100. Provide first aid within the scope of your training while waiting for professional help to arrive. Be prepared to describe accurately the nature of the accident.

Use of Emergency Equipment

Everyone working in DMSE laboratories must know how to use emergency equipment such as spill kits, safety showers, and eye wash apparatus. Know where these items are located in your laboratories.

Thermal Burns

- If the burn is minor, apply ice or cold water.

- In case of a clothing fire, the victim should drop to the floor and roll, not run to a safety shower. A fire blanket, if nearby, should be used to smother the flames.
- After flames are extinguished, deluge the injured areas under a safety shower. Keep the water running on the injured areas for 15 minutes to remove heat and to wash off chemicals.
- Place clean, soaking wet, ice-packed cloths on burned areas, and wrap to avoid shock and exposure.
- Do not use a CO2 fire extinguisher on a person with burning clothing; this could cause suffocation or frostbite. Dry chemical extinguishers will create inhalation hazards and contaminate wounds. Pressurized water can aggravate burn injuries.

Chemical Burns

- For chemical burns or splashes, immediately flush with water.
- Apply a stream of water while removing any clothing that may have been saturated with the chemical.
- If the splash is in the eye, flush it gently for at least ten minutes with clear water.
- Wash in a direction away from the other eye.
- If the splash is on the body, flood it with plenty of running water.
- A shower, hose, or faucet should be used in an emergency.
- For chemicals spilled over a large area, quickly remove contaminated clothing while using the safety shower. Seconds count, and no time should be wasted for the sake of modesty.

Traumatic Shock

- In case of traumatic shock, or where the nature of the injury is not clear, keep the victim warm, lying down, and quiet.
- Wait until medical assistance arrives before moving the victim.
- Report all injuries to your supervisor and the MIT Safety Office.

BASIC ELEMENTS OF LABORATORY SAFETY

Better than responding correctly to an accident, of course, is not having one in the first place. The following list of safety procedures is intended to keep them from happening, and these procedures are mandatory practice for all laboratory situations unless modified by the Laboratory Supervisor. Laboratory workers should consult the Supervisor if they feel these rules should be relaxed or tightened in particular cases.

Planning experiments

- Plan ahead: Seek information and advice about hazards. Plan appropriate protective procedures, plan positioning of equipment before beginning any new operation. Know what to do to prevent an accident and what to do if an accident occurs. Do not begin an experimental procedure until the Laboratory Supervisor has discussed these safety issues with you.
- Each laboratory worker must know the use and location of all first aid and emergency equipment in the laboratories, shops, and storage areas.
- Each laboratory worker must know the location of nearby telephones for summoning fire fighters, police, emergency medical service or other emergency response services. The emergency number (100) must be posted at many places throughout the building, and on each laboratory telephone.
- Each laboratory worker must be familiar with all elements of fire safety: alarm, evacuation and assembly, fire containment and suppression, rescue, and facilities evaluation.

Conducting experiments

- All injuries, accidents, and "near misses" must be reported to the Laboratory Supervisor. An Accident Report must be completed as soon as possible after the event by the Laboratory Supervisor.
- All chemical spills are to be reported to the Laboratory Supervisor, whose directions must be followed for containment and cleanup. Laboratory workers should follow the prescribed instructions for cleanup and decontamination of all spill areas.
- Protect your ears. The healthy ear can detect sounds ranging from 15 to 20,000 hertz. Temporary exposure to high noise levels will produce a temporary hearing loss. Long term exposure to high noise levels produces permanent hearing loss. There appears to be no hearing hazard (although there are possible psychological effects) to noise exposures below 80 dB. Exposure above 130 dB is hazardous and should be avoided. Ear muffs offer the highest noise attenuation and are preferred for levels above 95 dB. Ear plugs are more comfortable and are applicable in the 80-95 dB range. If you suspect that a hearing hazard exists, notify Environmental Medical Services and get the sound level measured.
- Unattended operations that could be hazardous should be avoided. When such operations must be conducted, the Laboratory Supervisor must approve the experiment, and the following precautions should be considered: leave lights on; place an appropriate sign on the door that includes the names(s) and phone number(s) of personnel that can be contacted in an emergency; and provide for containment of toxic substances in the event of failure of a utility service (such as cooling water, ventilation, electrical power, etc.).
- Do not work alone when conducting hazardous procedures, so that someone is available to summon help if the need arises. Some laboratories, especially teaching laboratories, will require that no work be conducted when alone.
- If you are working alone at times other than normal working hours, you may wish to notify Campus Patrol (3-1212) of your location and activities so that a patrol officer can check frequently as to your safety and locate you if an emergency occurs or you should require emergency assistance.
- "Horseplay" is hazardous and will not be tolerated.
- Long hair and loose items of jewelry or clothing must be secured during work with rotating machinery.
- Each laboratory worker must be familiar with an approved emergency shutdown procedure before initiating any experiment.
- No deviation from approved equipment operating procedures is permitted.
- All laboratory aisles and exits must remain clear and unblocked.
- Obsolete and unused equipment and materials must be removed from the laboratory, either to storage or disposal.
- The instructions on all warning signs must be read and obeyed.
- Good housekeeping must be practiced in the laboratories, shops, and storage areas.
- Only chemicals (no food) may be placed in the laboratory refrigerators, which should be "laboratory safe." Ice from laboratory ice machines may not be used for human consumption or to cool any food or drink.
- Avoid eating or drinking in laboratory areas where laboratory chemicals are used or stored; hands should be washed before conducting these activities.
- Smoking is not permitted in MIT facilities.
- Handle and store laboratory glassware with care to avoid damage; do not use damaged glassware. Use extra care with Dewar flasks and other evacuated glass apparatus; consider shielding or wrapping them to contain chemicals and fragments should implosion occur. Use equipment only for its designed purpose.
- Glassware breakage and malfunctioning instruments or equipment should be reported to the Laboratory Supervisor. There will be no open flames or heating elements used when volatile chemicals are exposed to air.

- Personal items brought into the laboratory should be limited as much as is practical to those things necessary for the experiment.
- Safety laboratory practices prohibit the presence of young children and babies in areas that have a potential for exposure to radioactive materials, toxic or hazardous chemicals, infectious agents, or where the children are exposed to possible injury from a laboratory or other type of accident.
- Casual visitors to the laboratory are to be discouraged and must have permission from the Laboratory Supervisor to enter. All visitors and invited guests must adhere to all laboratory safety rules. Adherence is the responsibility of the person visited.

Compressed Gas Cylinders

- Compressed gas cylinders must be secured at all times. Proper safety procedures must be followed when moving compressed gas cylinders. Cylinders not in use must be capped.
- Do not use grease on gauges or connections on compressed gas cylinders.
- Only gauges that are marked "Use no oil" are used for oxygen cylinders. Do not use an oiled gauge for any oxidizing or reactive gas.
- Laboratory workers are never to play with compressed gas hoses or lines or point their discharges at any person.
- Do not use adapters or try to modify any gas regulator or connection on compressed gas cylinders.
- All gas cylinders are to be returned to the proper vendor. Some small lecture bottles are the non-returnable type which become a disposal problem when empty or near empty with a residual amount of gas. When ordering gases in lecture bottle size, be sure to order the gases in a returnable cylinder.

CHEMICAL HYGIENE

General Rules

Chemicals can have devastating effects on exposed workers, and chemical hygiene must be given special attention. DMSE has a *Chemical Hygiene Plan* describing these dangers and procedures for avoidance in detail. This section of *The DMSE Safety Primer* is condensed from that larger document, and provides an introduction to chemical safety. However, the *DMSE Chemical Hygiene Plan* must be easily accessible in all DMSE laboratories, and laboratory workers must read and understand those portions of it that pertain to their own situations. Additional information is available online from the MIT Industrial Hygiene Office. The following general precepts should be followed by all laboratory workers for essentially all work with chemicals:

Knowledge of Hazard

- Lists of hazardous chemicals have been compiled by the Occupational Safety and Health Administration (OSHA), SARA, NIOSH and other agencies. These lists have been reviewed by the Industrial Hygiene Office and by the Departmental Chemical Hygiene Office; a modified compilation is provided in the DMSE Chemical Hygiene Plan. Principal Investigators must establish standard operating procedures for the use of any of the hazardous chemicals listed in Table 1 of that document.
- The identification and classification of hazardous chemicals used in each laboratory are the responsibility of the Laboratory Supervisor, and the Supervisor is also responsible for authorizing use of the chemical to individual laboratory workers.
- Each research worker using a chemical is responsible for knowing the particular hazards associated with use of that chemical. This information is contained on the Material Safety Data Sheets (MSDS) prepared by the chemical manufacturer, and includes the worker's Permissible Exposure Limit (PEL) to the material as well as other safety aspects. MSDS's for many chemicals are available from the MIT Safety Office and the Chemical Engineering Department Library, and

many of these are also available from the Internet (click here). The MSDS should be obtained from the supplier when new chemicals are purchased.

- Based on the information in the MSDS and the way in which the chemical is to be used, the research worker must be aware of the control methods that are required. These control methods encompass storage, use, and any disposal methods other than the normal pick up of waste chemicals by the MIT Safety Office. The research worker should review the safety data and proposed control methods with the Laboratory Supervisor.
- Under the direction of the laboratory Supervisor, each worker in the laboratory is responsible for proper storage, use, and disposal of all chemicals used by that worker, according to the control methods established as described in above. Any worker accepting laboratory space accepts responsibility for all chemicals in it. If other chemicals are found in the space, the Supervisor should be informed immediately.

Accidents and spills:

- For emergency assistance dial 100.
- Plan ahead for spills - read the MSDS for the appropriate procedure, and have the necessary equipment for cleanup and first aid on hand.
- Eye Contact: Promptly flush eyes with water for a prolonged period (15 minutes) and seek medical attention.
- Ingestion: Contact the MIT Medical Department (phone 3-4481) to determine initial actions and seek medical attention.
- Skin Contact: Promptly flush the affected area with copious amounts of water and remove any contaminated clothing. Seek medical attention.
- Cleanup: Promptly clean up small chemical spills when appropriate expertise, protective apparel, equipment and proper disposal resources are available to safely accomplish the task. For emergency assistance dial 100 and report the incident to the Dispatcher. It is MIT policy that the person who creates a spill is responsible for the cleanup.
- If you spill mercury, clean it up as quickly as possible by collecting the drops with a suction aspirator. Vent the area well and do not let the mercury touch your skin: the metal and its vapor are toxic. Sprinkling sulfur on the spilled mercury reduces the vapor pressure of mercury by formation of a skin of sulfide, but the mercury must still be collected with an aspirator.

Avoidance of "routine" exposure:

- Avoid unnecessary exposure to chemicals by any route.
- Skin contact with chemicals should be avoided as a cardinal rule.
- Unless part of an approved protocol, do not smell or taste chemicals. Apparatus which may discharge toxic vapors/gases (vacuum pumps, distillation columns, etc.) should be vented into local exhaust devices.
- Inspect protective gloves for tears, pinholes, etc. before use.
- The Permissible Exposure Limits (PELs) of OSHA and the Threshold Limit Values (TLVs) of the American Conference of Governmental Industrial Hygienists should not be routinely exceeded. The MIT Environmental Medical Service can provide information on any established PELs and/or TLVs.
- Prevent or minimize the release of toxic substances in cold rooms and warm rooms, since these have contained recirculated atmospheres.
- Prevent or minimize the release of toxic vapors and gases in most biological safety cabinets since these generally exhaust air directly to the laboratory through only a particulate filter.
- Reducing the potential for exposure to particularly hazardous chemicals can be achieved by restricting the use of the material to a designated area equipped with the proper control devices. This designated area can be a glove box, fume hood bench, or an entire laboratory depending on the manipulations required. Particularly hazardous substances are stored, used, and prepared for disposal only in designated areas.

Choice of chemicals:

- The use of the following five chemicals is illegal in the City of Cambridge, MA, and must not be brought onto the MIT campus. These chemicals are:
    - Soman GD - nerve agent
    - Lewisite - blister agent
    - Mustard HD - blister agent
    - VX - nerve agent
    - Sarin GB- nerve agent
- The signature of the principal investigator is required to purchase the following chemicals:
    - Ethyl Alcohol (Tax free alcohol)
    - Explosives
    - Hypodermic Needles and Syringes
    - Liquefied Petroleum Gases (LPG)
    - Nitrous Oxide Gas
    - Poisons
- Before a substance is received, information on proper handling, storage and disposal should be known by the user. No laboratory chemical should be accepted without a label that identifies the chemical's name, and an accompanying Material Safety Data Sheet.
- Use only those chemicals and/or quantities of chemicals for which the quality of the available engineering controls (e.g. chemical hood and ventilation system) is appropriate.
- Assume that all substances of unknown toxicity are toxic and minimize exposure to such substances as much as possible.

Personal protection:

- All containers must be labeled as to content, composition, and appropriate hazard warning: flammable, explosive, corrosive, toxic, etc. The laboratory worker's name and the date the container was filled must be on the label.
- Toxic chemicals will be exposed to the air only in a property ventilated hood. Flammable chemicals will be exposed to the air only under a properly ventilated hood or in an area which is adequately ventilated (airborne concentration will be less than the Permissible Exposure Limit ("PEL") specified by the appropriate OSHA standard).
- When airborne concentrations of chemicals are or could be of concern, consult the MIT Environmental Medical Service.
- The user should keep personal protective items clean. In case the user knows or suspects that the item has become contaminated, it should be promptly removed and cleaned prior to reuse. Any skin area that may have become contaminated should be promptly and thoroughly washed.

Use of a Hood:

- A chemical hood should be used for operations which might result in significant release (e.g. above the OSHA permissible exposure level) of toxic chemical gases, vapors or dusts.
- As a rule of thumb, consider the use of a hood or other local ventilation device when working with any appreciably volatile substance of unknown toxicity or with an airborne occupational exposure limit below 50 parts per million (ppm).
- Adequate hood performance should be confirmed before use. This can be done by checking the Vaneometer, warning light or checking with a piece of tissue. For the best chemical hood performance the user should keep the work area five or six inches behind the plane of the sash, keep the hood sash closed except when adjustments within the hood are being made, keep materials stored in hoods to a minimum and not allow such items to block or interfere with airflow. If you suspect that the hood is not working properly, contact Physical Plant (phone FIXIT).
- The hood should be kept "on" with the sash down when it is not in active use if toxic substances are stored in it, or if it is uncertain whether adequate general laboratory ventilation will be maintained when it is "off."

Waste Disposal:

- A label indicating "Chemical Waste" with the chemical name and concentration should be placed on each container of chemical waste by the user.
- The MIT Safety Office should be called to collect such chemical wastes and for answers to chemical waste disposal questions.
- Do not discharge to the sewer flammable liquids, acids or bases (unless the pH has been adjusted to a range from 6 to 10 and heavy metals are not present), toxic, malodorous, or lachrymatory substances or any substances which might interfere with the biological activity of the wastewater treatment plant, create fire or explosive hazards, cause structural damage or obstruct flow.

Protective Clothing and Equipment

- The Laboratory Supervisor will determine the type of personal protection needed in specific laboratories. The requirements will be posted clearly, and workers will be diligent in adhering to the guidelines.
- Eye protection worn when working with chemicals should meet the requirements of the American National Standards Institute (ANSI) Z87.1. This means that chemical safety goggles available from the Warehouse should be worn whenever there is a potential for chemical contact such as a liquid splash. When working with more than 10 ml of a corrosive liquid, a face shield should also be worn. Covering safety eyewear is equally important to the wearer of contact lenses. Additionally, employees are encouraged to inform their supervisors when contact lenses are worn, and medical personnel treating the individual in case of chemical contact, so that proper eye irrigation can be provided. For general laboratory work without the potential for chemical contact or splash, the routine use of goggles or safety glasses with side shields should be considered.
- When working with corrosive liquids, gloves made of a material known to be resistant to permeation and degradation from the corrosive chemical should be worn. For example, a neoprene glove provides excellent resistance against 10% nitric acid while an industrial latex glove provides only good resistance. With 70% nitric acid the same neoprene glove provides only good resistance and the use of an industrial latex glove is not recommended by the manufacturer. The MIT Safety Office can provide additional information on the chemical resistance provided by different gloves and protective clothing items.
- A laboratory coat should be worn when conducting laboratory activities when contamination is possible in order to reduce the potential for chemical contact and to protect street clothing. When significant potential for liquid contact exists the use of safety goggles, impervious gloves and an impervious apron over the laboratory coat should be considered.
- When working with allergenic, sensitizing, or toxic chemicals, gloves should be worn that are resistant to permeation by the chemical and inspected by the user for the absence of pin holes.
- Whenever exposure by inhalation is likely to exceed the airborne limits described in the Material Safety Data Sheet (MSDS) a chemical hood should be used; if this is not possible consult with your supervisor and/or the Environmental Health and Safety Office before doing any such work.
- Carefully inspect all protective equipment before using. Do not use defective protective equipment. Keep protective equipment clean.
- Laboratory users should ensure that they have in their laboratory an eyewash unit connected to the potable water supply. This eyewash unit should be operated periodically (at least quarterly) by the user to verify proper operation. Keep electrical wires/equipment away from t he area of the eyewash.
- Laboratory users should know the location of the nearest emergency shower.

Housekeeping

- Access to emergency equipment, showers, eyewashes, and exits should never be blocked by anything, not even a temporarily parked chemical, housekeeping or maintenance type cart and/or construction material.
- Original labels on containers of chemicals must be protected so that the identity of the contents and the hazards those contents present is known. When chemicals are transferred from the original container to a secondary container, a new label should be attached that shows the chemical

name(s). In any event, at the end of each workday, the contents of all unlabeled containers should be labeled or are to be considered wastes and placed into a properly labeled waste container. If unlabeled containers of chemicals are discovered, properly label the container if the contents are known, or call the MIT Safety Office so that the material can be properly identified and disposed.
- All chemicals should be placed in their assigned storage areas at the end of each workday.
- All working surfaces and floors should be cleaned regularly. Always, consider the measures that should be taken to prevent injury to personnel entering the laboratory to clean, collect waste, repair or remove equipment, etc.

Procedure-Specific Safety Procedures

Any written laboratory procedures should include a written description of the specific safety practices. Workers should read and understand these practices and requirements before commencing a procedure. Specific additional safety procedures follow, in this section, for the laboratory use of chemicals that may present special hazards.

Procedures for Carcinogens and Highly Toxic Chemicals

For their protection laboratory workers must follow the additional procedures described in this section when performing laboratory work with any select carcinogen, reproductive toxin, substance that has a high degree of acute toxicity, or a chemical whose toxic properties are unknown (when using or handling amounts greater than a few milligrams to a few grams, depending on the substance).
Prior to ordering a "special chemical" the Laboratory Supervisor should determine how to comply with these additional safety requirements. The Laboratory Supervisor must specify the designated area(s) and post the boundaries clearly. Only those persons trained by the Laboratory Supervisor to work with the "special chemical" and informed of its toxicity should use the substance. Such work should be done in the designated area. All users of special chemicals should conduct their work in accordance with the principles outlined below:

- Use the smallest amount of chemical that is consistent with the requirements of the work to be done.
- Minimize personal exposure by the consistent use, as appropriate, of a chemical hood, properly selected gloves, safety goggles, and laboratory coat that is removed by the individual prior to his/her leaving the laboratory.
- Use high-efficiency particulate air (HEPA) filters or high-efficiency scrubber systems to protect vacuum lines and pumps.
- Work on a spill containment tray and/or absorbent pad to facilitate cleanup and decontamination in case of a spill.
- Prepare for disposal any wastes from work with "special chemicals" as recommended by the Safety Office.

Procedures for Flammable Chemicals

In general, the flammability of a liquid is determined by its flash point, the lowest temperature at which an ignition source can cause the chemical's vapor to ignite momentarily in air under certain controlled conditions.

- Liquids with a flash point below 100oF (37.8oC) will be considered "flammable liquids."
- OSHA standards and the National Fire Protection Association (NFPA) guidelines apply to the use of flammable liquids in the laboratory. Consultant advice on these Fire Safety Regulations is available from the MIT Safety Office.
- Quantities of flammable liquids in the laboratory should be kept to a minimum consistent with laboratory needs and fire code mandates. Flammable liquids or items from which flammable vapors can evolve (e.g. ether) must not be stored in refrigerators/freezers, since most are not explosion- proof or explosion-safe.
- Flammable liquids should normally be used only in well ventilated areas away from sources of ignition.
- Special fire hazard potentials should be assumed to exist whenever oxygen is in use and/or oxygen concentrations in air are elevated above normal levels.

- Always store flammable liquids away from oxidizers.
- Be aware that liquids with flash points at and above 100oF may also present a significant fire hazard in case of ignition.

Procedures for Reactive Chemicals

Reactive chemicals are substances which may enter into violent reactions with the spontaneous liberation of heat and/or gases too rapidly to be safely dissipated. This may result in the rupture of the container, an explosion, fire or the release of toxic gases/vapors.

Laboratory users should handle reactive chemicals with all proper safety precautions, including segregation in storage. For example, nitric acid (a good oxidizer) should not be stored with flammables. Water reactives should not be stored in a location where the item could get wet. Users should not mix for the first time even small quantities of such reactive chemicals with other chemicals without prior approval of the Laboratory Supervisor.

For hot perchloric acid digestions, use only a perchloric acid hood, or use special scrubbers approved by the MIT Environmental Medical Service. This is necessary because the condensation of hot perchloric acid vapors inside the hood can result in the formation of explosive compounds that are shock sensitive.

Picric acid is useful for revealing grain boundaries and carbides in steels; however, it becomes highly explosive when it crystallizes out of solution during long-term storage (picric acid anhydride is an explosive). Therefore, the solution should be discarded within one week of preparation. If picric acid crystals must be used, procedures should be established to keep the crystals moist with water. Dry picric acid crystals are a shock sensitive explosive.

Some chemicals on aging form reactive compounds. For example diethyl ether forms peroxides that may be violently explosive. Thus, ether has an expiration date. Limit quantities of such materials and have a notification system so that outdated quantities of ether are collected by the MIT Safety Office.

Procedures for Corrosive Chemicals and Contact-Hazard Chemicals

Corrosive chemicals are those substances that, by direct chemical action, are injurious to body tissues or corrosive to metals. Users of corrosive liquids should take special precautions so that direct contact does not occur.

Hydrofluoric acid (HF) is an important example of this class of chemicals. HF is worth particular attention because it is being used in several departmental laboratories, including a teaching laboratory, and because it has some unusually dangerous features. It is both corrosive and a toxic chemical that is absorbed quickly through the skin. Serious injury or death may follow exposure even in cases where the victim is not aware of a chemical burn.

Local first aid in the laboratory in the case of almost all chemical exposures is confined to washing, eye washing, and safety showers. The exposed person must then go immediately to the Medical department. Hydrofluoric acid is the exception. After the initial washing, HF Antidote Gel (Calcium Gluconate) must be applied immediately and massaged into the affected area. Then go to the Medical Department. HF Antidote Gel is available from the Environmental Medical Service (3-5360). This gel must be available within easy reach of the user, and the instructions must be read before this acid is used.

The clean up procedure requires that slaked lime be used for neutralization. This forms calcium fluoride, which is not soluble in water. The neutralized slurry should then be collected with an absorbent spill pad, and placed in a container for disposal by the MIT Safety Office.

Ventilation

Work with significant quantities of toxic chemicals that have low air concentration limits (Threshold Limit Value less than 50 ppm), or that have high vapor pressures, should always be done in a hood. At nights and weekends laboratory general ventilation is reduced and users should place special emphasis on performing in chemical hoods all operations that might release significant amounts of chemicals and/or contact Physical Plant (phone FIXIT) so that their general ventilation needs can be met.

Chemical hoods should provide a minimum face velocity of 90 feet per minute (average) at any working height that will be used unless a different face velocity is approved by the MIT Environmental Medical Service.

Laboratory employees should understand and comply with the following principles:

- A chemical fume hood is a safety backup for condensers, traps, or other devices that collect vapors and fumes. It should not be used to "dispose" of chemicals by evaporation unless the vapors are trapped and recovered for proper waste disposal. For a chemical fume hood to provide significant protection it must be used and maintained properly by the user;
- The work or apparatus inside the hood should be placed at least six inches behind the sash;
- The fume hood sash should be closed at all times except when necessary to adjust the apparatus that is inside the hood (when hoods have horizontal sliding panes, the panes should be kept closed);
- The hood fan should be kept "on" whenever a chemical is inside the hood, whether or not any work is being done in the hood;
- Personnel should be aware of the steps to be taken in the event of power failure or other hood failure (e.g. stop work, cover chemicals, close hood, notify Supervisor);
- Physical Plant and the Environmental Medical Service inspect hoods at periodic intervals to be sure they are working properly.
- Hoods should not normally be used as storage areas for chemicals, apparatus, or other materials.

Environmental rooms are NOT well ventilated and procedures carried out in such rooms should be carefully designed to minimize personal exposures.

Flammable Liquid Storage

Flammable liquids in quantities greater than 500 ml should be kept in flammable liquids storage cabinets. If such flammable liquid storage cabinets are not available, the flammable liquids should be kept inside cabinets and not left on the floor or counters. When flammable storage cans are used, never disable the spring-loaded closure. Always keep the flame-arrestor screen in place; replace the screen if it is punctured or damaged. Flammables should not be stored with incompatible materials like oxidizers or in refrigerators and freezers since most are not explosion-proof or explosion-safe.

Cabinets designed for the storage of flammable materials should be properly used and maintained. The user should read and follow the manufacturer's information and should also follow these general safety practices:

- Store only compatible materials inside a cabinet;
- Do not store paper or cardboard or other combustible packaging material with flammable liquids;
- The manufacturer establishes quantity limits for various sizes of flammable-liquid storage cabinets; cabinet should not be overloaded.

Eyewash Fountains and Safety Showers

All laboratories have been provided with an eyewash connected to the potable water system. Safety showers are located in the hallways. Users need to know the location and how to operate such devices. Users need to periodically flush and check the functioning of their eyewash fountains and make sure that electrical wires and devices are clear of the eyewash. This should be done on at least a quarterly basis. Facilities Management periodically checks the emergency showers and verifies proper operation. Users are encouraged to report problems with such safety devices promptly to Facilities Management for evaluation and repair.

Be sure that access to eyewash fountains and safety showers is not restricted or blocked.

Respirators

- Persons requiring respirators to protect against chemical exposure must contact the MIT Environmental Medical Service, which will assist in:
    - Selection of the respirator;
    - Fit testing of the respirator;
    - Training on the use, care and limitations of the respirator; and
    - Employee medical certification to wear a respirator.
- Surgical masks are not to be used to provide respiratory protection against chemical overexposure.
- The wearing of contact lenses with full-face respirators is not permitted under OSHA regulations.

Vapor Detection

Odor should not be relied upon as a means of determining that inhalation exposure limits are or are not being exceeded. Whenever there is reason to suspect that a toxic chemical inhalation limit might be exceeded, whether or not a suspicious odor is noticed, notify the supervisor and/or the Environmental Medical Service. As an interim measure, laboratory use of the chemical should be stopped, or the use of the chemical limited to a chemical hood.

Chemical Waste Disposal

MIT Policy

The proper disposal of waste chemicals at the Institute is of serious concern, and every effort should be made to do it safely and efficiently. The responsibility for the identification and handling of waste chemicals within the Institute rests with the Supervisor in whose laboratory the waste was created, and the supervisor must budget for the cost of pickup and disposal. A procedure for waste disposal should be planned before a project is started. Wastes must be labeled properly. Inadvertent mixing of incompatible materials must be avoided.

Storage Area

The Institute has provided a storage area for waste chemicals; the waste is accumulated here until there is sufficient quantity to justify transportation to a disposal site. The Safety Office maintains this storage area, and the only access is via the Safety Office.

Transportation

A pick-up of waste chemicals may be arranged by calling the MIT Safety Office (X3-4736). The person creating the waste is responsible for transporting the containers of waste to the storage area when pick-up service is not available.

General Procedures for Waste Disposal

Plan a procedure for waste disposal before you start on a project. Label waste properly. It is up to each department, group, or experimenter to identify waste materials properly before disposal; inadvertent mixing of incompatible materials could have serious consequences. Analysis to determine the identity of unknown chemicals is very expensive, and these costs will be borne by the laboratory supervisor.
Protection of the environment makes the disposal of large quantities of chemical and solid wastes a difficult problem. It is in everyone's best interest to keep quantities of waste to a minimum. The following suggestions may help:

- Order as small a quantity of material as practical, even if you can get twice as much for the same money.
- Use only the amount of material that is needed for conclusive results.
- Avoid storing excess material, particularly if it is an extremely toxic or flammable material, just because you may want it in the future.
- Before disposing of unwanted, unopened, uncontaminated chemicals, check with others in your department who may be able to use them.
- When a researcher completes a research project or a thesis, he or she must label all unused chemicals to be kept by the laboratory.

Make sure all samples and products to be disposed of are properly identified, labeled with their chemical names, and containerized. You must clean up before you transfer within or leave MIT. You must submit a Departure Compliance Form to the DMSE Safety Office before you transfer within or leave MIT. For more information on identifying waste, see the subsequent sections on "Identification," "Unknown Waste," and "Paperwork."

Procedures For Specific Waste Categories:

- Organic solvents must not be put down the drain. Regulations that apply to MIT's sewer system prohibit the discharge of organic solvents to the sewer system. This applies to all organic solvents

whether flammable or nonflammable, miscible or non miscible with water. Organic solvents should be placed in suitable containers (1 gallon maximum) where there is no danger that vapors or the liquid will escape. Containers shall be capped tightly, labeled prominently, and picked up by the MIT Safety Office .

- Mixtures of organic solvents that are compatible and combined in one container must be identified with an estimated proportion in fractions or percentages of each solvent in the mixture indicated.
- Many laboratory operations create neutralized acids and-alkaline solutions which may be put down the drain provided that they do not contain heavy metals or toxic contaminants. Concentrated acids and caustics, acids and alkaline solutions should be put into proper containers tightly capped, sealed with laboratory film such as "Parafilm M", labeled, and given to the Safety Office.
- Inorganic and organic solids in their original containers that are contaminated, old, or of questionable purity may be given to the Safety Office.
- Mercury must be removed from lab apparatus and put into jars or bottles before sending it to the MIT Safety Office. Broken mercury thermometers must be put into a jar or secondary container. Clean-up materials from a mercury spill may be containerized, labeled, and sent to the Safety Office. Any laboratory or department that is interested in sending mercury waste to be distilled and to receive a credit for the mercury must take the responsibility of getting the mercury waste to the proper vendor.
- Cyanide compounds, arsenic, lead, and heavy metal wastes should be placed in bottles and containers, sealed tightly, labeled, and given to the Safety Office.
- Alkali metals such as sodium and potassium should be placed in a suitable container, covered with Nujol (mineral oil), labeled properly, sealed so that there is no possibility of their coming into contact with water, and given to the Safety Office.
- Pyrophoric metals such as magnesium, strontium, thorium, and zirconium, and other pyrophoric chips and fine powders should be placed in a metal container, sealed tightly, labeled, and given to the Safety Office.
- Waste oil in quantities of less than 1 gallon may be sent to the waste oil chemical storage area or given to the Safety Office. Large quantities of waste chemicals to be removed from a laboratory may be more than a normal amount for the Safety Office to pick up, and the Laboratory Supervisor will be financially responsible for the disposal. Some examples are the wastes collected in drum lots from a research project, the clean-out of a laboratory of old reagents and chemicals which would be packed into drums, and the waste chemicals to be pumped out of a collection or storage tank.
- Transformer oil which may contain PCB's should be tested for PCB content. The responsibility of having the transformer oil tested and for the actual disposal rests with the department involved.
- Capacitors that contain PCB's are likewise the responsibility of the department involved. Information on possible disposal contractors can be obtained by calling the MIT Safety Office (X3-4736).
- Equipment containing PCBs should not be accepted in transfer from other institutions or from other departments within MIT. If you accept PCB-containing equipment, you also accept a very large toxic waste disposal bill that only escalates with the passage of time.
- Controlled drugs to be disposed of as waste must not be sent to the waste chemical storage area. The handling, recording, and disposal of controlled drugs are the responsibility of the department involved operating within the Bureau of Narcotics and Dangerous Drugs (BNDD) Regulations.
- Biological waste that may contain live viruses must not be sent to the waste chemical storage area. The disposal of biological wastes is handled in accordance with procedures for deactivation that have been established by the department involved and the Environmental Medical Service.

The Environmental Medical Service may be consulted if there is any question concerning the toxicity or packaging of any toxic wastes.

Identification

All waste chemicals must be identified by chemical name, including the proportions of a mixture. All containers must be labeled prominently because the safe transportation of chemicals is possible only when everyone who handles the containers knows the identity of the contents.

Unknown Waste Chemicals

Unknown waste chemicals cannot be accepted for disposal. Disposal contractors cannot accept or ship unknown waste. It is the responsibility of the Laboratory Supervisor involved to identify all chemicals; this may require polling laboratory personnel, students, and faculty members to ascertain the owner of such unknown waste and its identity. Ultimately, it may require the services of an analytical laboratory to analyze the waste. This can be dangerous particularly when opening containers of unknowns, so it must be emphasized constantly to laboratory workers to identify and label all waste chemicals and project products with a chemical name.

Packaging

Waste chemicals must be packaged and containerized in a manner which will allow them to be transported without danger of spillage, explosion, or escape of dangerous vapors. Wastes which have not been properly packaged and identified will not be accepted for disposal.

Paperwork

A packing list must be filled out by personnel in the laboratory or department that requests that the waste picked up by the MIT Safety Office . The packing list must be filled out with the quantity, chemical name, designation as a solid or liquid, and hazard associated with the waste, i.e., flammable, toxic, water-reactive, etc. Safety Office personnel will bring the packing list with them when they pick up waste chemicals.

RADIATION SAFETY

A number of acute and long term effects on humans have been related to exposure from various types of ionizing radiation. Radiation hazards arise when using radio-isotopes, lasers, x-ray generators and plasma torches. Each is hazardous in a unique way. A thorough knowledge of the device or the isotope that is to be used is mandatory. The precautions vary widely. Information pertaining to the particular hazard should be obtained from the facility prior to use, or from the Radiation Protection Office of the Environmental Medical Services. However, several precautionary procedures should always be followed:

- All work with radioactive material or equipment that produces ionizing radiation must be registered with the Radiation Protection Office of EMS and performed in accordance with the MIT Required procedures for Radiation Protection.
- Review with the Radiation Protection Office any potential exposures to non-ionizing radiation such as ultraviolet, visible, infrared, and microwave radiation.
- Clearly mark areas in which lasers, radiation, and ultraviolet or high intensity light sources are in use. Standard signs are available from the Radiation Protection Office.
- Wear appropriate eye protection when working with these sources.
- Be aware and alert to radiation hazards when working in or visiting a laboratory where radiation is generated.
- Class IIIb and class IV lasers require a written Standard (Safe) Operating Procedure (SOP), and registration with the MIT Radiation Protection Office.

ELECTRICAL DEVICES

Electricity is in constant use both within and outside the laboratory, so it is easy to forget that significant physical hazard or death may result from its misuse. With direct current, a male can detect a "tingling" feeling at 1 mA and the median "let-go" threshold (the current at which he cannot release the conductor) is 76 mA. For 60 Hertz alternating current, the values are 0.4 mA and 16 mA respectively. Women are more sensitive to the effects of electrical current than males; approximately 2/3 of the above currents is needed to produce the same effect ("Electrical Hazards 5.1," Technical Information, MIT Safety Office). Higher currents produce respiratory inhibition, then ventricular fibrillation, and ultimately cardiac arrest. Although minute electrical shocks are generally considered annoying rather than harmful, such shocks constitute an ominous warning of the presence of potentially hazardous conditions. The device in question should be disconnected immediately and the cause ascertained by a person competent in such matters.

Work on electrical devices should be done only after the power has been shut off in such a manner that it cannot be turned on accidentally. Internal current-carrying devices such as capacitors must be discharged. All "home-made" electrical apparatus should be inspected and approved by someone competent in electrical circuitry before being placed in service.
Observe the following rules when working with electrical equipment:

- Ungrounded wiring and two-wire extension cords are prohibited. Worn or frayed extension cords or those with broken connections or exposed wiring must not be used. All electrical devices must be grounded before they are turned on.
- Extension cords are for temporary use, and are not to be used n place of permanent wiring.
- Volt Limit: Untrained persons may not work on live equipment carrying potentials greater than 50V.
- Use only tools and equipment with non-conducting handles when working with electrical devices.
- If you feel an electrical "tingle" while working with a piece of laboratory equipment, disconnect it and consult with your supervisor. In the U.S., three-terminal (115 V AC) electrical wiring should conform to the following color code:
    - White = neutral wire
    - Black = live/hot wire
    - Green = ground wire

  (When working with existing wiring, do not trust that this color scheme has been used correctly.)
- Do not short circuit the leads to a battery. Without a fuse, the internal resistance of the battery will cause it to heat and possibly explode. Dangerous arcs or flashes may also be produced.
- A ground-fault interrupter does not assure protection against electrocution.
- All current transmitting parts of any electrical devices should be enclosed.
- When checking an operating circuit, keep one hand either in a pocket or behind the back, to avoid grounding yourself.
- Maintain a work space clear of extraneous material, such as books, papers, and clothes.
- Never change wiring with the circuit plugged into a power source.
- Never plug leads into a power source unless they are connected to an established circuit.
- Remove rings, watches, or other such jewelry before working on electrical circuits.
- Avoid contacting circuits with wet hands or wet materials.
- Wet cells should be placed on a piece of non-conducting material.
- Check circuits for proper grounding with respect to the power source.
- Do not insert another fuse of larger capacity if an instrument keeps blowing fuses. This is a symptom requiring expert repairs.
- Keep the use of extension cords to a minimum and cords as short as possible. Tie off excess cord out of pathways.
- Do not use or store highly flammable solvents near electrical equipment.
- Multi-strip outlets should not be used in place of permanently installed receptacles.
- Keep access to electrical panels and disconnect switches clear and unobstructed (three feet of floor space).
- Make certain that all electrical equipment (lamps also) is properly grounded.
- Be alert and aware of the dangers inherent in high voltage equipment.

In the event of a small electrical fire:

- Turn off the power source and unplug the equipment.
- Do not turn on the circuit until the cause of the fire has been established and the fault corrected.
- Report the fire to the Safety Office.

CRYOGENIC SAFETY

Handle any liquefied gas carefully. At these extremely low temperatures, these gases can produce an effect on the skin similar to a burn. Eyes should be protected with a face shield or safety glasses. Gloves should be worn. Stand clear of the boiling and splashing liquid and its issuing gas. *Should any liquefied gas contact the skin or eyes, immediately flood that area of the body with large quantities of unheated water and then apply cold compresses.*

Oxygen is removed from the air by liquid nitrogen. Therefore, use liquid nitrogen only in a well-ventilated area so that the ambient oxygen concentration does not drop lower than 19.5% (same for liquid helium).
The high pressure gas hazard is always present when cryogenic fluids are used, since these are usually stored at the boiling point. Never obstruct the vent valve on cryogenic containers. Wood or asphalt saturated with liquid oxygen has been known to explode when subjected to mechanical shock.
When using a liquid nitrogen cold trap, charge the trap only after the system is pumped down. Otherwise, considerable amounts of liquid oxygen could condense, thus creating a major hazard.
An excellent source of reference, which is strongly recommended for anyone working with cryogenic materials is *Safety with Cryogenic Fluids*, Michael G. Zabetakis, Plenum Press, New York, New York, 1967.

FIRE SAFETY

Emergency Procedures

Do *not* try to fight a fire yourself if you are uncertain of being able to handle it; call for help.
If a fire starts, call for assistance by pulling the nearest fire alarm box, and evacuate the building (do not use the elevators). **DIAL 100** from a safe location and give what information you have. Do not return to the building unless permitted to do so by the Fire Department.
If your clothes ignite, "stop, drop, and roll" to smother the flames. Do not run; running only intensifies the flames. When fire blankets are readily available, use them to wrap around yourself to aid in putting out the fire.

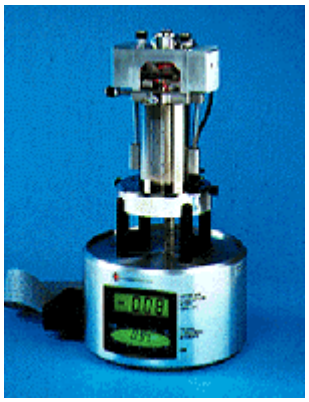
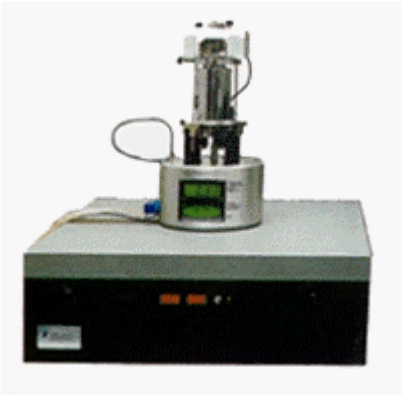
Precautionary Procedures

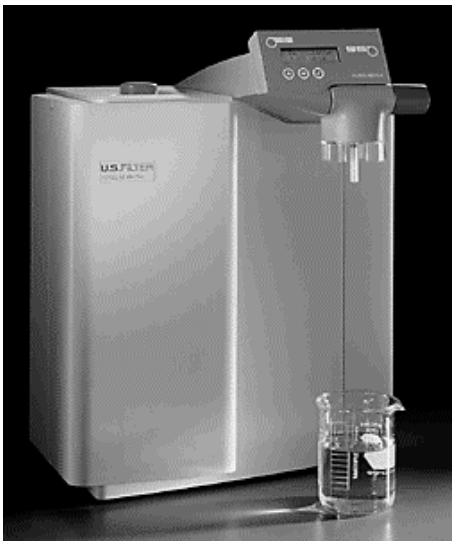
Know the location of fire exits, fire alarm pull stations, fire blankets and extinguishers. Each laboratory should be equipped with an extinguisher or extinguishers. Fire extinguishers are primarily for fire fighters.
In rooms where the air flow is high, a natural gas leak could occur undetected. If there is one or more fume hoods in a lab, check for leaking valves by brushing a soapy liquid around the valve stem and over exit hole. If you see any bubbles in the liquid, call Frederick Wilson (13-4078/X3-6866) and Physical Plant (FIXIT) to report the leak.
Keep all fire doors closed at all times.
Do not block access to fire escape routes.
Neatness prevents many fires. Fire spreads much faster when it has cluttered waste materials to feed on.
Oily rags, waste, or papers improperly stored are important causes of spontaneous combustion. Store these materials in covered metal containers.
The informational document *Guide to Classes of Fires* provides some guidance on firefighting methods.

7. LABORATORY NOTEBOOK GUIDELINES

- Each section has a clear, descriptive heading of the experiments which were performed and detailed writing of the experimental observations, thoughts, and results.
- Each entry is dated.
- Each entry is legible.
- Each entry is in English.
- Each entry is written immediately after the work was performed.
- Multiple lab notebooks should be labeled numerically in the order of which they were written.
- Experimental data, originals or copies (e.g. micrographs), should pasted or taped into lab notebooks. If copies are made, originals should be organized in a separate binder on which the lab notebook number and page are denoted.
- On collaborations, clearly indicate who did what work and who was present for which experiments.
- At the end of a research project, all lab notebooks will be returned and archived in the group for use by as reference for future students.

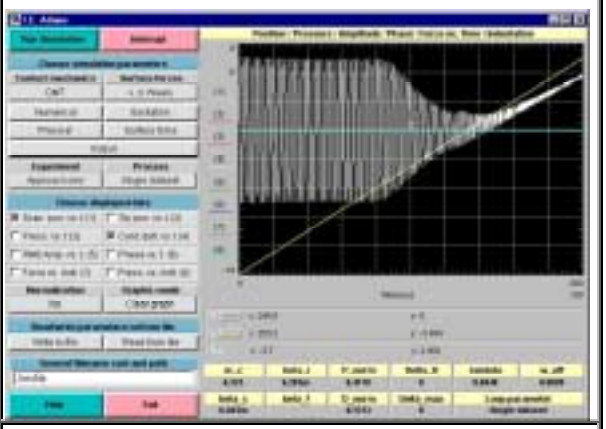
8. DESCRIPTION OF LABORATORY EQUIPMENT

Molecular Force Probe (MFP) *Asylum Research, Inc. :* • Brief description of MFP and MFP specifications from Asylum Research, Inc. • MFP Saftety Precautions • Power Spectral Density of Deflection Thermal Noise of 0.01 N/m Cantilever taken with MFP in Air and Water • *Thermomicroscopes* Microlever Specifications and Corresponding Force Noise Levels with MFP *(*courtesy of J. Cleveland, Asylum Research, Inc.)* • Instructions for Statistical Analysis of High Resolution Force Spectroscopy Adhesion Data	
Atomic Force Microscope (AFM): • *Digital Instruments* NanoScope® IIIA System Controller with Multimode™ AFM • DI Multimode Saftety Precautions • Procedures for Imaging of Standards at Atomic-Scale Resolution Using the Digital Instruments Multimode AFM • Instructions for Converting of Raw Multimode High Resolution Force Spectroscopy Adhesion Data *(*courtesy of M. Rixman)*	
Halcyonics MOD-1 Active Vibration Isolation System	

PureLab Plus UV/UF Laboratory Water Purification System (US Filter)	
AT200 Analytical Balance (Mettler-Toledo)	
PerpHecT pH Meter (ThermoOrion)	

Nanomechanics Software :

•*IC Adams* Nanomechanics Software



Optical Microscopes :

• *Axioskop 20,* Zeiss, Inc. with transmitted and reflected light, differential interference contrast, cross-polarizers, incident light flourescence.

• *Axioplan* Zeiss, Inc. with transmitted and reflected light, differential interference contrast available through the MIT CMSE shared facilities

Digital Camera:

• *KODAK Digital Science Microscopy Documentation System 100 (MDS 100)*

Micro- and Macro- Mechanical Testing Equipment :	
• *Rheometric Scientific (Polymer Labs)* Minimat 2000 Miniature Materials Tester with Environmental Chamber • *DMS 110 SEIKO* and *DMS 200* Dynamic Mechanical Rheology Station available through the MIT CMSE shared facilities	

9. MFP SAFETY PRECAUTIONS

Ortiz Nanomechanics Lab : 13-5037

1. **PHOTODIODE AND LASER KNOBS :** Please be sure not to push them beyond their range of motion. They are quite sticky at the ends and may get stuck or the belt may come off.

2. **FLUIDS.** Although there is minimal danger if fluids leak down into the base, please be careful when inverting the head and keeping the tip wet. If some of the fluid leaks back into the "volcano" passing through the tip holder / laser focusing lens into the depth of the volcano-it can damage all of the main MFP components.

3. **WIRE CONNECTING HEAD TO BASE**: Be careful not to twist or stretch it too much when changing samples/tips.

4. **LASER FOCUSING LENS** : Please do not use the laser focusing lens, there is no safety guard on it [Summer, 20000].

5. **BUNGEE TRIPOD :** Please do not use with MFP or anything similar, there is a pretty big danger of it becoming unbalanced and falling off.

10. MULTIMODE AFM SAFETY PRECAUTIONS

Ortiz Nanomechanics Lab : 13-5037

I. TIPHOLDERS :

• Only use the AFM tipholders provided with the Multimode**, do not use any early-model AFM tipholders** from older AFM's, they may short out the power supply.

II. LASER :

• During and prior to laser alignment, **avoid looking directly at the laser beam**, the laser spot, or reflected laser light-especially with highly reflective samples. Staring at the laser beam can cause eye damage.

• **Do not manipulate highly reflective samples while the laser in on.**

• **Never plug in the laser unless the head is secured** on top of the base with the piezo springs.

III. FLUIDS

• **Do not operate the standard tipholder in fluids.** Standard tipholders have exposed electrical signal lines that could cause a short circuit if exposed to a conducting fluid.

•**When imaging in fluids use extraordinary precautions against spillage on any component of the AFM.** Fluids must not be spilled on or around the sample stage, electronic

boxes, or other components containing electronic parts. Avoid spilling all corrosive fluids on exposed surfaces; otherwise damage will result (e.g. shorting out of piezo tube scanners). In case of a spill, immediately clean and dry all affected surfaces thoroughly.

- **Do not overfill the fluid cell.**
- **Do not allow solvents to splash on the scanner tube or wiring at the center of the scanner body**: certain components (e.g. wiring insulation) may be dissolved, causing scanner failure.

IV. PIEZOS

- **On the scan control panel, avoid Offset voltages outside of +/-150V for periods of more than one hour.** When possible reset them to zero. Piezo crystals can be damaged by having large DC offset voltages applied to them for long periods of time. In the **Realtime/Scan** controls panel there are parameters called **X offset** and **Y offset**. The voltages which correspond to these values can be seen by selecting **Units : Volts** in the **Other Controls** panel. (NOTE: the **X offset** and **Y offset** will change when a **Zoom In/Out** or **Offset** command is used).
- **Keep the Z-center near zero volts.** The offset voltage on the z-piezo (line-by-line average) is displayed by **the Z-center position** indicator bar on the Display monitor. It should also be kept low (between +/- 150V) when possible. After engagement, it can be adjusted by using the **Motor / Step Motor** command. (Tip Up goes more positive, if nothing happens, keep clicking. If necessary, increase the Step Size to overcome mechanical backlash). Another means is the

Withdraw and then re-engage again. Thermal drift or other reasons may cause the z-center to change gradually with time. It change suddenly when if tip leaves the surface resulting in a fully retracted piezo (-220V).

• **Avoid using maximum scan sizes for long periods of time.** Some crystals have sensitivities which will allow them to scan much larger than their nominal size specification. Running a crystal near maximum scan size over many days will result in a gradual lowering of sensitivity, which will change the calibration. The slight decrease will tend to level out as the crystal ages.

V. USE OF STIFF CANTILEVERS / TAPPING MODE

• **Use Force Mode with extreme precaution.**
With stiff cantilevers (e.g. Si tapping mode air tips), force mode can potentially damage both the tip and sample.

• **Choose the setpoint as low as possible.**

• **Before enabling the interleave drive amplitude, check that this value is not much greater than the main drive amplitude** to prevent tip damage.

VI. SOFTWARE / HARDWARE

• **Never leave the controller ON while the computer is turned off.** The DSP (Digital Signal Processor) and Interface boards inside the computer are extensions of the Nanoscope Controller. They must be energized to maintain the Nanoscope controller at proper outputs. Without the

computer, outputs to the piezo might be at random values (up to +/- 220V).

- **Do not unplug cables to / from energized hardware. Turn OFF first.**
- **Do not insert a conducting object into the phase extender box while it is energized** (e.g. screwdriver).
- **When closing the System.par file after viewing, if you are asked to SAVE CHANGES, be sure to say NO.**

VII. ENGAGEMENT / IMAGING

- **Never attempt to manually engage using the course adjustment screws.**
- **Ensure engagement settings never exceed safety limits**
- **Never move or adjust the AFM head while imaging or taking force curves.**

11. FORCE CURVE DATA CONVERSION
(M. Rixman)
For data from the DI Multimode AFM

1. Export the raw data as an ASCII file

Do this in the Offline/Utility menu. Header information is known to cause problems in some third party software applications and should therefore not be included in the ASCII file. *Be sure to save the raw data file.* The header information will be required for the data processing, and can be accessed from the raw data file.

2. Obtain necessary parameters from the header file

Required parameters include:

(from the *Scanner list):	\@Sens. Zscan (nm/V)
(from the *Ciao scan list):	\@Sens. Deflection (nm / V) (from
the (from the *Ciao force list):	Scan Rate (Hz)
	Z scan start (V)
	Z scan size (V)
(from the *Ciao force image list):	Samps/line
	\@Z scale (V / LSB)

3. Open the ASCII file and begin data conversion from LSB to nm

The data will probably be in one long column of a length equal to twice the number of samples per line. If Samps/line=512, the first 512 values are the extension values and the second 512 values are the retraction values. The data points are in the order that you would view them on the actual force curve from left to right. For ease in data analysis, it is best to separate these into two adjacent columns. If you have converted the force curve into something other than the default (deflection vs z-piezo position), such as force vs separation, then there will be one column of a length equal to *four* times the number of samples per line; the first two sets of data will be the default values (i.e. deflection vs z-piezo position), and the second set will be the converted values (force vs separation, e.g.). In each set, the extension data will precede the retraction data, and will be ordered as you would view them on the force plot from left to right.

The data points are in LSB (8-bit two's-compliment) format, and must first be converted into A/B voltages and then into nanometers before further analysis may be done. The process is outlined below:

$$A/B\ voltage = \{data\ point\ (LSB)\} \bullet \{\backslash @\ Z\ scale\ (V/LSB)\}$$

$$deflection = \{A/B\ voltage\ (V)\} \bullet \{\backslash\ Sens.\ Deflection\ (nm/V)\}$$

Now that you have converted the data points on the ordinate of the force curve, you must calculate the corresponding z-piezo position data points. Begin by converting Z scan start and Z scan size from V to nm by multiplying them by the \@Sens. Zscan value (nm/V). The first deflection point in both the retraction and extension data sets corresponds to the piezo position at Z Scan Start. Each successive deflection data point is reported after

a determined distance from the previous z-position has been traveled. The Z distance between data points may be calculated as follows:

$$Z\ dis\tan ce\ between\ data\ \ po\text{int}\ s = \frac{Z\ scan\ size\ (nm)}{Samps/line}$$

The raw ASCII data has now been converted and is ready for further analysis.

4. Convert the *deflection vs z-piezo position* data into *force vs separation*

Begin by plotting the raw data; there is some key information to be gotten from the plot. First locate the jump-to-contact point of the extension curve. Offset *all* of the data points such that the initial point of contact is at the origin of the graph.

The slope of the retraction curve *to the left of the point of pull-off* is the *sensitivity* of the piezo. Divide all deflection data points by this value; the resulting values are the actual deflection data:

$$\delta\ (nm) = \frac{raw\ data\ \ po\text{int}\ (nm)}{retraction\ curve\ slope}$$

Next convert the *z-piezo position* values into *separation from the surface* by subtracting each of the retraction and extension δ values from their corresponding z-piezo data points:

$$Separation\ (nm) = Zpiezo\ \ position - \delta$$

**NOTE: If you wish to plot the deflection vs z-position curve before proceeding to conversion to force vs separation, e.g., the above calculations should be modified such that the deflection is found by dividing the raw data points by the *negative* of the retraction curve slope. When calculating separation, then, you should *add* rather than subtract the deflection value from the z-piezo position. Doing so will give you the desired curves in both plots.

Finally, convert the deflection data into force data by multiplying each of the points by the spring constant k (in N/m) of the cantilever tip used to make the measurement:

$$Force\ (nN) = \delta \times k$$

And *voila*! Plot the force vs separation and you may obtain the forces involved in the jump-to-contact and the pull-off. Over a series of force curves of the same sample a statistical analysis if these forces may be performed to obtain well-supported experimental information on your sample. In addition, the slope of a line connecting the last point of adhesion to the first point after pull-off should be equal to the spring constant of your cantilever tip.

12. INSTRUCTION FOR CONDUCTING STATISTICAL ANALYSIS ON HIGH-RESOLUTION FORCE SPECTROSCOPY ADHESION DATA

1. Open the raw experiment *.PXP datafile of interest in Igor Pro.
2. On the first drop down menu, go to File/Open File/Procedure and then open the procedure file **RecordCursor.ipf** (the source code will open). This procedure file will allow you to record the values of any particular datapoint you like from each force curve into a table using the cursors.
3. Go to the drop down menu Macro/Compile. The drop down menu should now have two additional options. Prepare for Record Cursor and Record Cursor.
4. Hit Macro/Prepare for record cursor. A 3 column table will appear in which the data will be recorded.
5. Hit MFP display/Make review graph and the first force curve will appear. If you would like to record data from this force curve, hit modify on the review panel, zero force and hit LVDT/Deflection to convert raw data into converted force versus tip-sample separation distance.
6. Select data point of interest using cursor and then go to Macro/Record Cursor. The datapoint will be entered into the table. If you are recording discrete binding forces, you need to take two datapoints (middle of upper value and middle of lower value) to get the relative value. You should do this for each one for consistency so that you can subtract them later in excel. Repeat for every datapoint you would like to record.
7. Copy data in your table and import into peakstats.txt Excel file. (If you need to do the subtraction to get the relative values for binding forces, do it in Igor before hand). Paste special (values) into the G column of peakstats. Copy the peak force data in nN. Paste special (values) into H column of peakstats. Place an X at the bottom of the columns. Save this file under another name.txt and close it.
8. Open the distribution.xls excel file
9. Enable Macros.
10. check if in the correct directory . File; open (the name.txt file that you just saved should be visible).
11. Tools; macro; macros; highlight distribution; run.
12. In “filename” type the name of the file created in (7) – example: procedure.txt
13. In “Intervals (Peak Magnitude, nN) type interval values. Examples: 0.001, 0.002, 0.005 (nN)
14. In “Intervals (Peak Distance, nm) type interval values. Examples: 1, 3, 5 (nm).
15. Press continue. Wait. End at next pop-up.
16. The J& K columns created are the calculated force values. The V&W columns created are the calculated distance values. A total of three intervals are possible, thus intervals can be altered two more time and data put into the columns that follow.
17. Highlight the two columns from which you want to make the graph (i.e. J&K or V&W). Insert; chart; scatter XY; finish.
18. Chart; chart type; custom types; user-defined; probability.
19. Unhighlight data is necessary to move scale.
20. Save as a newly named.xls

13. PROCEDURES FOR IMAGING OF STANDARDS AT ATOMIC-SCALE RESOLUTION USING THE DIGITAL INSTRUMENTS MULTIMODE AFM

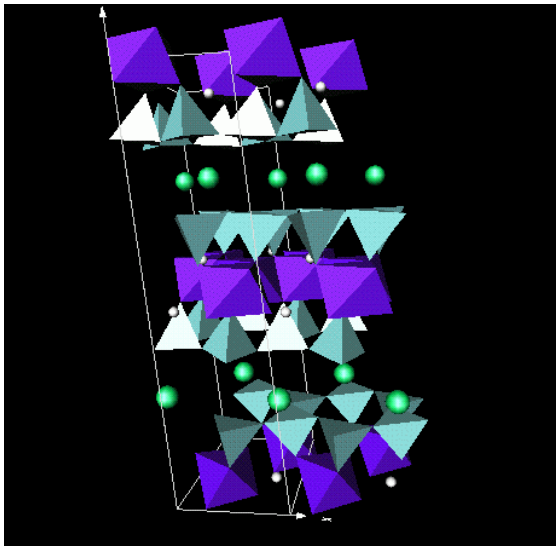
Ortiz Nanomechanics Laboratory@MIT F01

I. Sample Producers and Preparation

• **Muscovite Mica** is a yellowish, light-colored, transparent to translucent silicate (subclass : phyllosilicates) mineral with the following chemistry : $K_2Al_4Si_6Al_2O_{20}(OH,F)_4$, Potassium aluminum silicate hydroxide fluoride. It is a hard, layered, crystalline (monoclinic 2/m) material that fractures along weak atomic planes ("cleavage" planes), thus easily producing atomically flat surfaces of atoms having a regular lattice structure which are excellent for use in high-resolution AFM piezo calibration and as substrates for imaging of biological samples. Muscovite has a layered structure of aluminum silicate sheets weakly bonded together by layers of potassium ions. The potassium ions occupy large holes between 12 oxygen atoms, 6 from the layer above and 6 from the layer below; the resulting K-O ionic bonds are rather weak and easily broken. The cleavage sheets fairly flexible and elastic, hydrophilic, and negatively charged in water. Muscovite Mica has low iron content is a good electrical and thermal insulator. More detailed information on Muscovite Mica can be found here : *http://www.2spi.com/catalog/submat/mic_shet.html*

*(*http://www.ill.fr/dif/3D-crystals/layers.html)*

Another typical AFM substrate is **Highly Ordered Pyrolytic Graphite (HOPG)** which is described in detail here : *http://www.2spi.com/new/hopgsub.html*



• A few suppliers include the following companies :
  1) Bioforce Lab (*http://www.bioforce.com*)
  2) Structure-Probe Inc. (**http://www.2spi.com/catalog/afmstm.html*),
  3) Microscopy Mart / Pelco International *(*http://www.pelcoint.com/AFM.htm)*

• Cleavage directions are detailed here : *http://www.2spi.com/catalog/submat/mica-disk.html*

• Attach to metal puck and then to piezo scanner cap.

II. Real Time Parameter Settings

• contact mode, air or fluid cantilever holder (it is typically much easier to obtain images in water or buffer than in air)

• use stiffest spring constant cantilever; on DI V-shaped cantilever chips this is the shortest one with fattest legs

• use oxide-sharpened Si_3N_4 probe tips, e.g. Model NPS which has the following specifications (*http://www.di).com/products2/NewProbeGuide/ContactModeProbes.html* :

PARAMETER	VALUE
spring constant	0.58 N/m
nominal tip radius of curvature	5-40nm
cantilever leg length	100μm
cantilever configuration	V-shaped
reflective coating	Au
shape of tip	square pyramidal
tip half angle	35°

• use piezo scanner with smallest distance range available, A or EV scanner, E scanner should also work (the difference is the engagement mechanism).

• load sample, turn microscope and vibration table on, and then focus the laser as far out on the cantilever as possible to obtain the highest sensitivity

• let the system stabilize thermally for 30 minutes (with hood on)

• laser focusing (find maximum): the laser should never been switched off, i.e. turn the system on and off and all manipulations done with the laser on

PANEL	PARAMETER	SETTING
Scan Controls	Scan size	1 μm
	X offset	0.00 nm
	Y offset	0.00 nm
	Scan angle	0.00 deg
	Scan rate	61.00 Hz
	Number of samples	512
	Slow scan axis	enabled
	Z limit	between 55V-440V
Feedback Controls	Integral gain	0.001
	Proportional gain	0.00
	Lookahead gain	0.00
	Setpoint	0 V
Other Controls	AFM mode	Contact
	Input attenuation	1x
Interleave Controls	Interleave mode	Disabled
Channel 1	Data type	Height* (*setting this parameter to deflection is typically easier)
	Highpass filter	OFF, 3-4
	Lowpass filter	OFF, 1

III. Imaging

• **Engage** the surface. Make sure you are not false engaged (*see Section 10.10.1 of the DI AFM Manual).

• Reduce **Scan Size** to ~12 nm.

OR

• **Engage** with the **Scan Size** set to zero and slowly increase.

• Increase **Scan Rate** to 60Hz. Notice that if the **Scan Rate** is set much higher for atomic scale images to defeat some of the noise due to thermal drift.

• Adjust **Integral Gain**, **Setpoint, Scan Rate,** and **Scan Angle** to obtain a good image. Initially, the **Setpoint** should be kept as low as possible initially and then increased to obtain an image. The **Z-center position** should be close to 0V. The **Scan Angle** is known to have a huge effect, with optimal imaging conditions if the

sample is rotated until the atoms are oriented vertically or when the fast scan axis is parallel to the a or b crystallographic axis. The **Proportional Gain** should stay at zero except for large scan sizes (~70% of the scanner range). The system determines the minimum value of the **Integral Gain**. If you start with a value less than the system's minimum, you wont get an image.

•**Filters**, you should be able to obtain an image with the filters off. In general, the filters should be set to off since filtering during data acquisition affects raw data and height values.

• If difficulty is experienced obtaining and image **Withdraw** and try a different location on the sample surface, then **Engage** again. See Section 15.10.1 of the Multimode AFM Manual for Troubleshooting Contact Mode imaging.

• Once an adequate image of the surface is obtained (see Figures 1-4), make sure the image is real by varying the **Scan Size**. The spots observed should scale with **Scan Size**. The image you will obtain is based on the "stick-slip" frictional motion of the probe tip (which is why there needs a certain amount of force to be applied) determined by the spatial periodic corregations on the crystalline lattice surface.

• If you notice a bright vertical band on either end of the image, this is due to the abrupt reversal of direction of the scanner at the end of each scan line. You can eliminate this by using the following procedure :

1) select **Microscope / Calibrate / Scanner**

2) A window will open with the scanner calibration files and in the lower right hand corner is the "rounding coefficient" which is the percentage of the last portion of each scan line that is not displayed. This should initially be set equal to 0.0. Increase this to ~0.2 (while not exceeding 0.5) and this will cut off the last 20% of each scan line.

3) Reset this value back to 0.0 when finished.

• **Capture** the image.

• Sometime a good image can suddenly vanish, possibly due to adsorption of surface contaminants. Try different XY locations on the same cleaved plane and cleaving the sample a few other times.

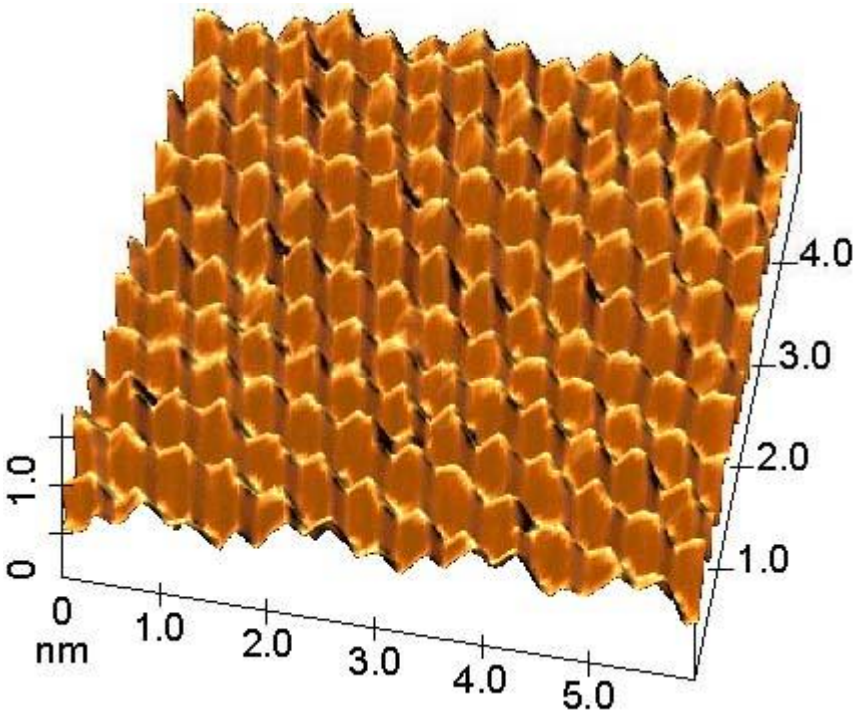


Figure 1. AFM High-Resolution Contact Mode Image of Mica from A.Belyayev, State Research Institute of Physical Problems & NT-MDT, Moscow, Russia.
(unpublished) (*downloaded from : *http://www.ntmdt.ru/scangallery/index.php?action=fullview&id=34)*

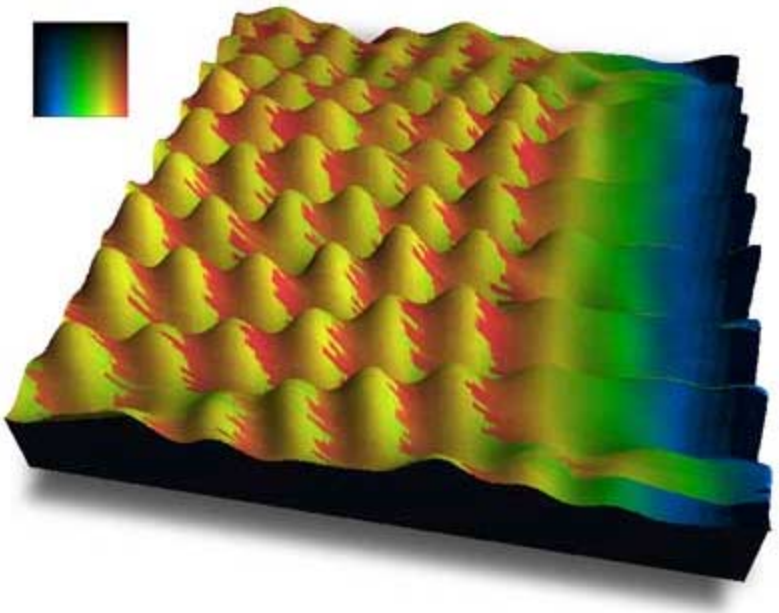


Figure 2. AFM High-Resolution Contact Mode Image of HOPG (*downloaded from : *http://stm2.nrl.navy.mil/how-afm/how-afm.html)*

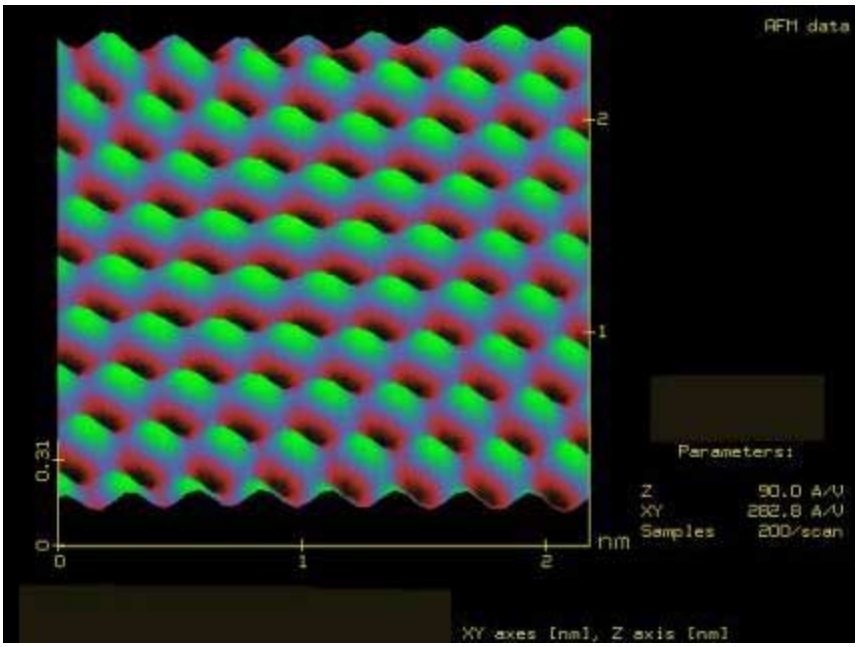


Figure 3. AFM High-Resolution Contact Mode Image of HOPG (*downloaded from : *http://www.physics.sfasu.edu/afm/afm.htm*)

(http://www.energosystems.ru/fgallery.htm)

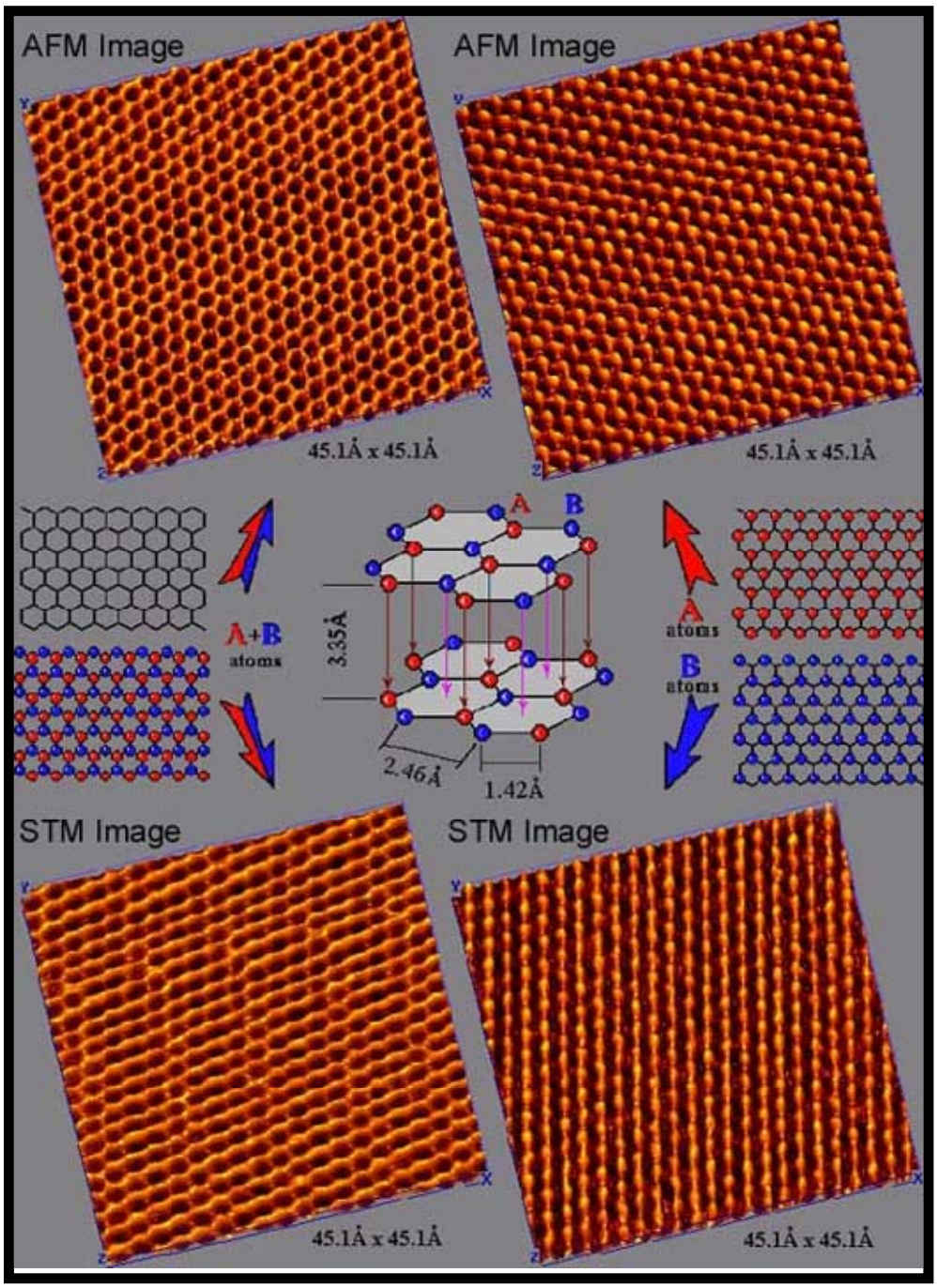


Figure 4. AFM High-Resolution Contact Mode Image of HOPG

IV. Off-line Image Analysis.

• Go to **Off-line / View / Top View** option and measure the spacings between atoms. The spacings should be as follows (as shown in Figure 5) :
MICA : A=0.519 nm, B=0.900 nm, C=1.37 nm
HOPG : A=0.255 nm, B=0.433 nm, C=0.666 nm

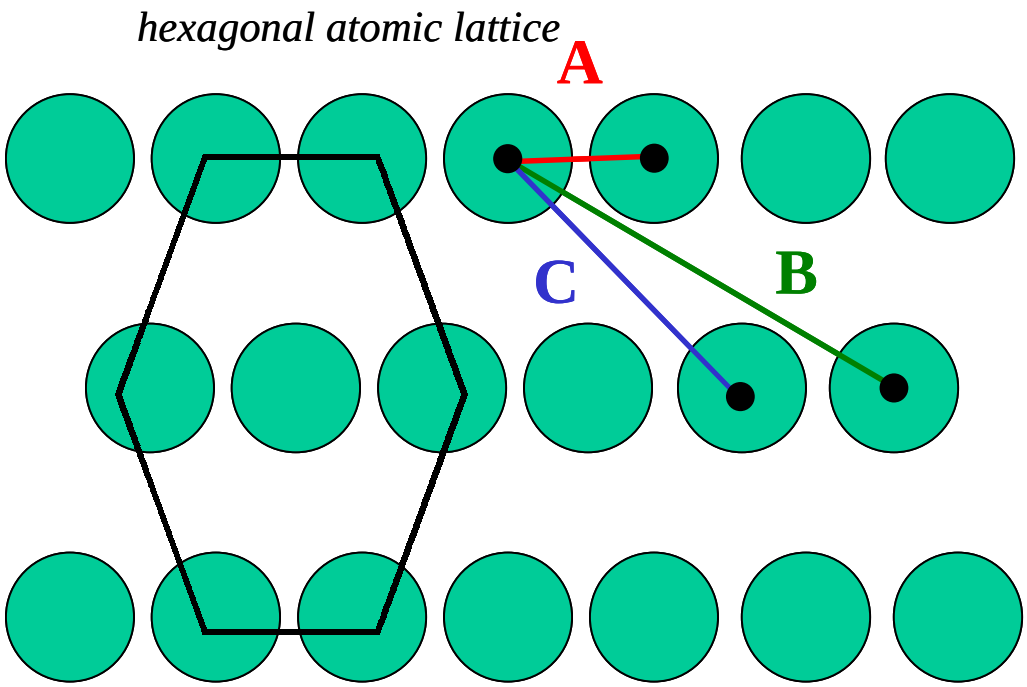


Figure 5. Hexagonal Atomic Lattice

Record the spacings for ~ 10 atoms observed in a captured image and average them. This can be done by alternatively "walking" the cursor line from atom to atom; the average distance will be shown on the bottom right hand corner of the display monitor's status bar. If the measurements vary by more than 2 percent from the dimensions shown above, a correction should be made as follows.

V. X and Y Axis Corrections

• See Sections 15-7.2-15-7.3 of the DI Multimode manual. The only difference is that the known distances must be adjusted for the smaller atomic spacings of the atoms. Furthermore, the sensitivity parameters are adjusted for atomic-scale imaging as follows :

PARAMETER
X fast sens 0° **Scan Angle**
X slow sens 0° **Scan Angle**
Y fast sens 90° **Scan Angle**
Y slow sens 90° **Scan Angle**

The derate parameters are not changed for atomic scale imaging including ; x fast derate, x slow derate, Y fast derate, Y slow derate, retracted offset der, extended offset der.
As stated in section 15-7.9 in the DI Multimode manual, the sensitivity parameters must be calibrated with the Scan angle set at both 0 degrees and 90 degrees. Z-axis calibration is done the normal way using a silicon calibration reference (see Section 15.8 of the Multimode Manual for detailed instructions).

VI. References

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4. Ohnesorge, *et al., Science* **260** *1451* (1993)**[(1014) cleavage plane of a calcite ($CaCO_3$) crystal]**
5. *Digital Instruments / Veeco Metrology Group* Multimode™ SPM Instruction Manual Version 4.31ce (copyright 1996-1999), Section15.9.
5. Encyclopedia Brittanica Online :
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14. TRAVEL AND REIMBURSMENT POLICIES

Research grants will cover the following for approved business travel :
1. direct transportation (e.g. flights, taxis, car rental)
2. housing (e.g. hotel)
3. conference fees and registration

All arrangements should be made in advance by the group member and also paid for by the group member. Original receipts, including flight ticket stubs, should be provided to Tim Doyle (12-009) soon after the conference (within one week) along with the title of the conference and explanation of the receipts. He will prepare the paperwork, send it to Prof. Ortiz for the approval signature, and then submit it through the MIT system for reimbursement which typically takes 2 weeks time. He will contact you when your reimbursement check arrives which you can pick up from him in 12-009.